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THE DEVELOPMENT OF AN AUTOMATIC MULTISAMPLE ISOTHERMAL AGING APPARATUS

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A prototype isothermal aging apparatus has been developed in which up to ten samples can be exposed to temperatures up to 1000°F for long periods of time. Weight losses of the samples are automatically determined at preset intervals and the information is stored on paper tape. The reproducibility of the data excellent. The apparatus is being adapted for computerization of the data.

FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division. The work was initiated under Project No. 7340, "Nonmetallic and Composite Materials," Task No. 734004, "New Organic and Inorganic Polymers." It was administered under the direction of the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with Dr. G. F. L. Ehlers (AFML/MBP) as Project Monitor.

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INTRODUCTION

The investigation of the behavior of polymers by determining weight losses during long-term exposures to elevated temperatures has in the past been hampered by relatively crude testing techniques. The test materials were usually placed in heating blocks or ovens and then periodically removed, weighed, and replaced. It is obvious that thermal histories of samples subjected to continuous heating and cooling cycles will differ significantly from those of a material kept in a controlled isothermal environment. The effects of cyclic environmental changes were partially circumvented by exposing numerous samples of the same material and successively withdrawing samples for a single weighing at a specified time interval. These samples were not returned to the oven so the opportunity to examine progressive weight loss on a specific sample was eliminated. This technique remains, moreover, a tedious and time-consuming one as well as being limited in a practical sense to either small sample populations or short test durations.

Another inadequacy found in some past experimental approaches to determining the resistance of materials to thermal environments has been the attainment of controlled temperatures above 600°F.

These shortcomings have now been overcome in the Laboratory by the development of a multisample isothermal aging apparatus in which ten samples (e.g., either nine single samples plus one reference blank or four samples and a blank in duplicate) can be exposed to controlled temperatures up to 1000°F for indefinite periods. The system provides for automatic weighings and data recording at preset time intervals. A print-out is obtained which includes weight, temperature, and elapsed time. It is planned to augment these automatic features by computerizing the handling and reduction of test data. Work to this effect is currently in progress.

The design, construction, and assembly of the apparatus was accomplished by Systems Research Laboratories, Dayton, Ohio under Contract F33615-72-C-0226. Final assembly including installation, testing, and instrumental modifications were performed subsequently at AFML/MBP. This report covers work performed during the period from December 1971 to December 1973.

SECTION II
APPARATUS

The system for measuring isothermal weight loss consists of a furnace containing the turntable, heater, and heating block; a thermo-balance with heat shield, hang-down wire, and pick-up; a master control console, including a Digital Voltmeter-print-out; and hardware for the vertical and circular movements of the turntable (Figure 1).

The furnace is constructed of an outer aluminum shell, Transite insulation, and an inner stainless steel lining and is designed for minimum heat loss under the test conditions. Some heat loss is unavoidable through the opening for the balance hang-down wire, however, the temperature control at 700°F is ±3°. The furnace has a working space of 5 x 7 x 7 inches, and contains an air inlet and a window for visual observation of the interior.

A cylindrical chrome-plated copper block located on top of a ring heater rests on the turntable which operates on a carousel-type principle similar to the one described in Reference 1. It is capable of maintaining a set of up to ten samples at a preset temperature, moving each sample in turn so that it can be weighed by the balance at fixed time intervals. The temperature is controlled by a stepless controller with a sensing range of 0-1000°F. The heating element is a circular heater with 1000 W output. The heating block contains indentations for ten samples. The movement of each sample is accomplished through a cam for up-down motion of the turntable and a pulley attached to a spline for rotary motion. Control of the motion is actuated through a relay circuit containing a series of micro switches. The master control contains the drive motor, relay circuitry, temperature - time control, and power switches.

The balance, a Cahn Electrobalance Model RM-2, has a total capacity of 5 gram, 3.5 grams of which are due to the tare consisting of the hang-down wire, the pick-up, and the specimen holder. This allows for a net sample capacity of more than one gram. The sample, usually 10 to

30 mg, is weighed in an aluminum cup and placed in the specimen holder. The latter is made of titanium as is the hang-down wire and pick-up. The hang-down wire is connected to the balance by means of a platinum friction loop which opens when stress is applied. This acts as a safety feature for the balance. The heat shield, a glass-aluminum plate, prevents the temperature at the balance orifice from rising above 130°F when the furnace temperature is maintained at 700°F, well within the temperature tolerances for the electrobalance.

The print-out provides a record of the weight to 0.1 mg, the (air) temperature in degrees F , and the elapsed time in days, hours, and minutes. The weighing interval between samples can be set between 2 and 30 minutes, permitting a range of from 20 minutes to five hours for the cycle of ten samples. Switches are provided (1) to correct any discrepancies between the sample number and the print-out number, (2) to choose between (three)thermocouples, (3) to adjust the actual number of samples by omitting any empty specimen holders, and (4) to switch from automatic to manual and vice versa (the timer is inoperative in the manual position). A manual print-out command switch is also provided as is a manual sample advance switch.

The air flow (40cc/min) is regulated with a flowmeter, and the air is predried by conducting it over Drierite and through concentrated H_2SO_4.

The apparatus is located on a sturdy wooden platform with leveling screws so that the horizontal position of the balance can always be checked and maintained. A photograph of the apparatus is shown in Figure 2.

The apparatus functions in the following manner: The turntable moves to position the sample holder such that when it is lowered the sample holder is engaged and suspended for weighing. After the weighing the turntable is raised, lifting the specimen holder off the balance pick-up. The turntable then moves forward so that the next specimen holder is properly positioned for suspension and weighing, etc.

The present model of the isothermal aging apparatus is the result of several modifications. These include a complete redesign of the specimen holders since the original ones were made of quartz and too fragile to avoid excessive breakages. The modified version was made of titanium and its shape and size redesigned to increase stability and lower the center of gravity. The cell indentations in the heating block were enlarged and the rims were sloped to improve the alignment, suspension, and seating of specimen holders. The pick-up and hang-down wires were modified to prevent twisting and swaying of the specimen holder. Extensive modifications in the circuitry were also necessary to prevent skipping, misprints, and other electronic malfunctions.

Safety features were also incorporated, such as cut-off switches which are activated if the door to the electric motor is opened or the control thermocouple is either shorted out or open. These features prevent uncontrolled overheating in case of thermocouple failure.

SECTION III
EXPERIMENTAL

Temperatures were determined both within and above the specimen holders. The latter position duplicates the location of the specimen while in the weighing position and also corresponds to the temperature recorded on the printout. In a full complement of specimen holders this position is held by each holder 10% of the total time and provisions were made to compensate for the temporary but considerable temperature difference between the turntable and the air surrounding it. The relationship between the temperature setting and the temperature within the specimen holder has been determined and is shown in Figure 3. The experimental results were compared with tests conducted manually in an aluminum block under closely controlled temperature conditions. There was good agreement between tests.

Polymer samples are weighed into aluminum cups which fit into the specimen holders. They are then dried overnight at 90°C and approximately 2-5 mm Hg and reweighed before being inserted into the preheated furnace. The heat is temporarily turned off to avoid overheating and the turntable is cycled manually to check the alignment. The air is turned on and the apparatus is switched to automatic. Since it usually takes from two to six hours before complete equilibrium is attained, the initial readings are usually somewhat erratic. These are subsequently corrected by extrapolation of the weight loss curve. The data are plotted as weight loss (mg) versus time (hours). Any mechanical or electronic irregularities are readily detected by comparison with the reference blanks. These data are then plotted as percent weight residue versus time, usually in 24-hour intervals. Some typical results are shown in Figures 4 and 5. A percent weight residue plot is shown in Figure 6.

The reproducibility of the apparatus is illustrated by the data in Table I. These represent four separate runs with a full complement of eight samples of the same material, an aromatic polyester. The last run differs from the previous ones in that the material was predried.

SECTION IV
CONCLUSIONS

Use of this apparatus will significantly improve the data quality and accelerate long-term characterization and evaluation of candidate materials for subsequent development in Air Force applications. In addition, it will eliminate most of the manpower hitherto needed to perform time-consuming repetitive operations commonly associated with isothermal aging techniques.

REFERENCES

1. J.M. Ferguson, R. J. Fuller, and D. Mortimer, "Thermal Analysis" (Proceedings Third ICTA Davos 1971), 1, 197. Birkhaeuser Verlag, Basel 1972.

TABLE I

WEIGHT LOSS DATA OF AN AROMATIC POLYESTER AT 700°F IN AIR

% Weight Loss

1st Run

SAMPLE	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs
1	10.9	21.0	30.4	39.9	50.0
2	9.6	19.2	28.3	38.0	46.5
3	8.4	18.5	26.9	34.9	43.7
4	9.2	19.8	28.6	37.3	46.1
5	8.1	16.7	25.2	33.8	41.4
6	10.1	19.6	28.4	39.2	48.0
7	9.8	18.6	29.4	40.2	49.0
8	9.4	18.1	26.9	35.6	44.1
$\bar{x}$	9.4$\pm$0.6	18.9$\pm$1.0	28.0$\pm$1.3	37.4$\pm$1.9	46.1$\pm$2.3
		2nd RUN			
1	10.2	21.1	32.1	44.2	52.4
2	9.9	20.3	30.2	41.9	49.4
3	11.2	22.3	32.5	44.7	52.3
4	9.4	19.4	28.8	40.8	48.3
5	10.0	20.1	30.6	41.1	47.9
6	9.1	19.1	28.2	39.8	47.7
7	10.8	18.8	27.3	37.7	45.0
8	10.6	21.2	31.0	42.3	49.6
$\bar{x}$	10.2$\pm$0.5	20.3$\pm$0.9	30.1$\pm$1.5	41.6$\pm$1.7	49.1$\pm$1.8
		3rd RUN			
1	10.6	20.4	31.2	41.5	51.7
2	9.3	20.2	31.1	41.4	51.3
3	11.3	20.7	30.8	39.9	50.0
4	9.8	19.5	28.3	38.9	48.7
5	9.5	19.5	29.5	39.4	48.5
6	10.6	20.8	30.6	40.8	50.2
7	10.0	19.9	29.5	39.5	49.1
8	9.3	19.3	29.2	39.2	49.2
$\bar{x}$	10.0$\pm$0.6	20.0$\pm$0.5	30.0$\pm$0.8	40.1$\pm$0.9	49.8$\pm$1.0
		4th RUN (Predried)			
1	8.9	17.8	26.0	34.9	44.4
2	7.9	16.9	25.4	34.4	43.4
3	8.1	16.2	24.8	32.9	41.4
4	8.4	17.3	26.2	35.0	43.9
5	9.5	18.6	27.8	37.3	47.6
6	8.6	17.1	25.7	34.6	42.9
7	8.3	17.3	25.6	34.3	43.2
8	8.8	17.3	25.7	34.5	43.0
$\bar{x}$	8.6$\pm$0.4	17.3$\pm$0.4	25.9$\pm$0.6	34.7$\pm$0.7	43.7$\pm$0.8

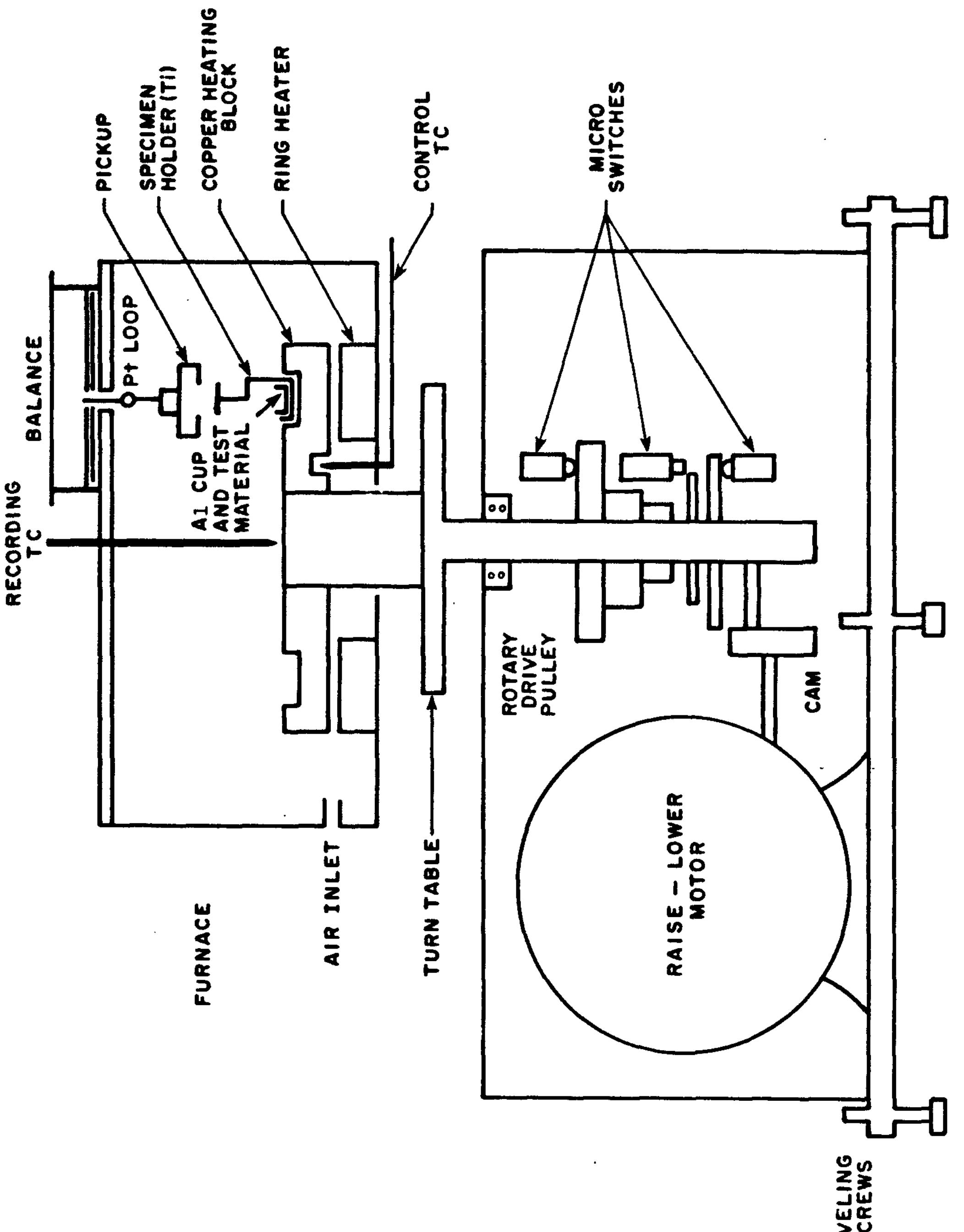

Figure 1. Isothermal Aging Apparatus, Hardware Schematic

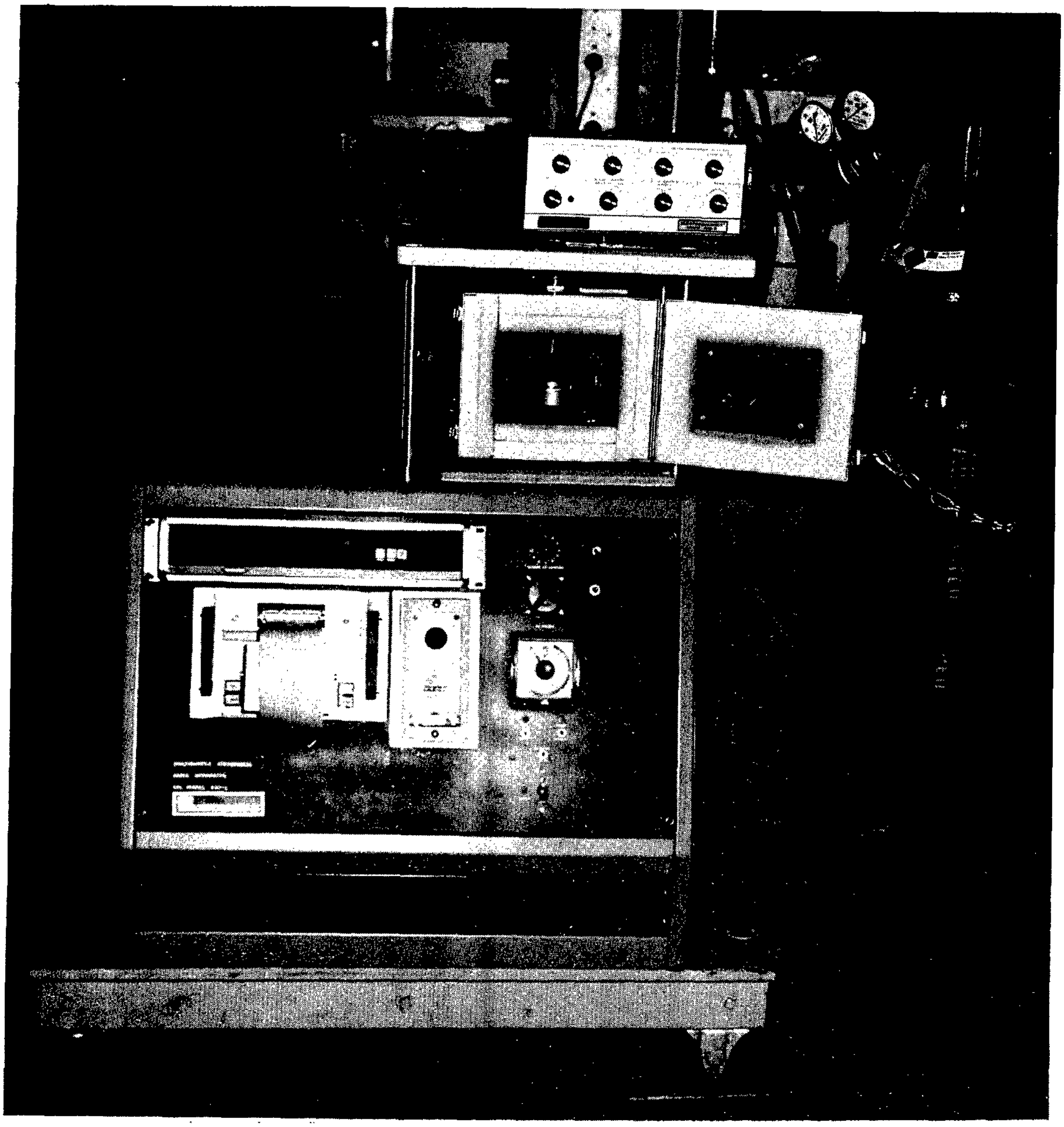

Figure 2. Isothermal Aging Apparatus

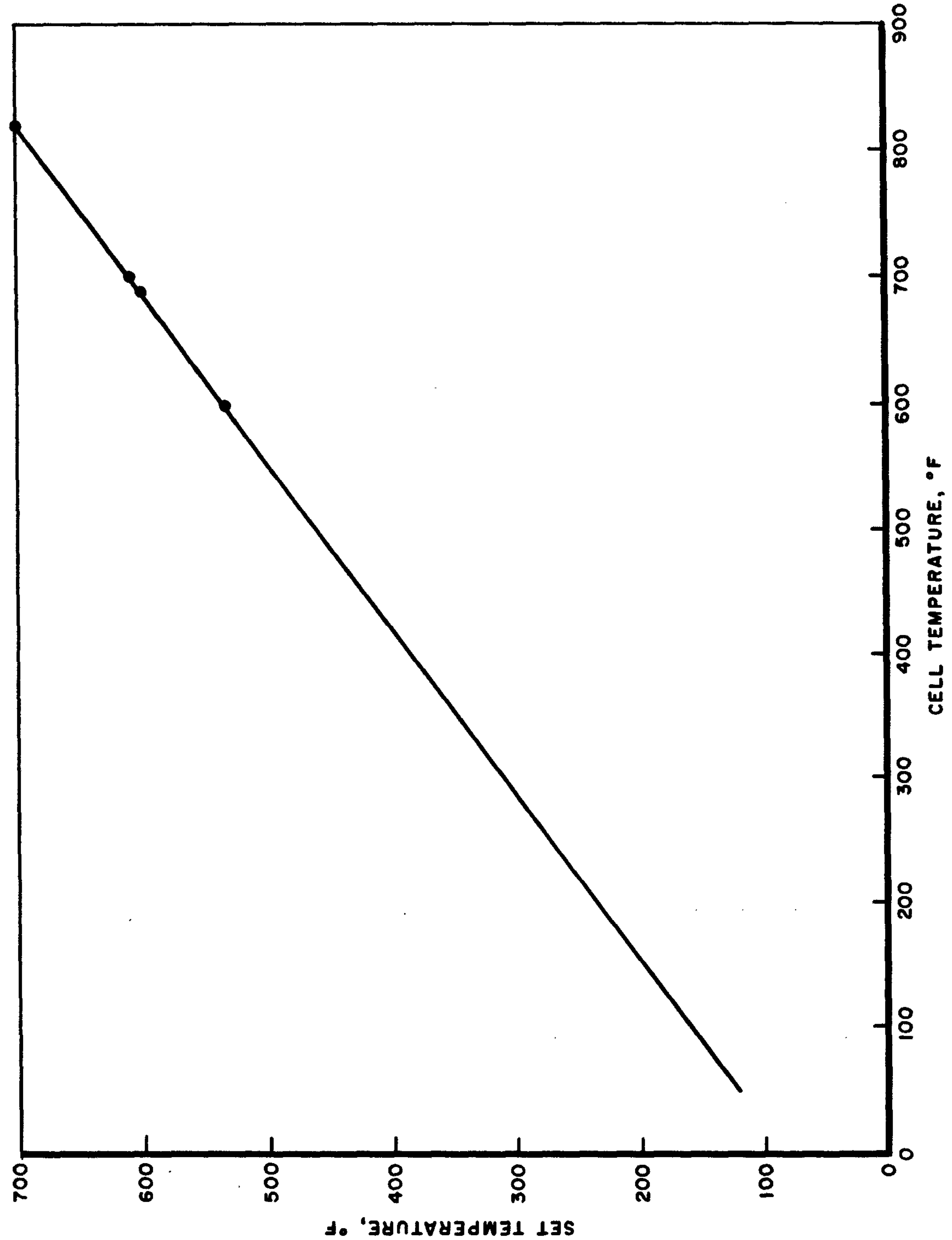

Figure 3. Cell Temperature versus Set Temperature

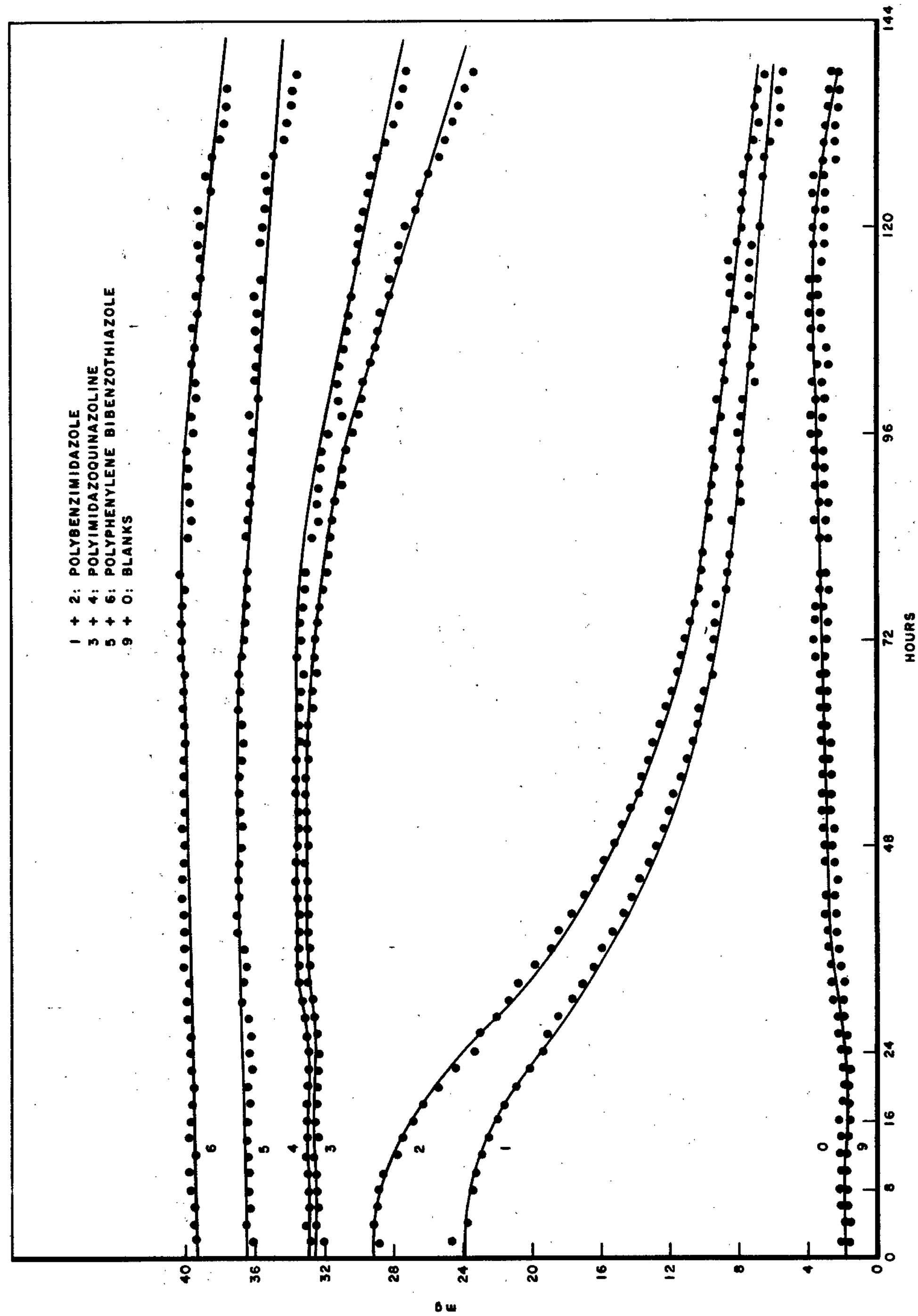

Figure 4. Weight Loss Curves of Several Polymers (I)

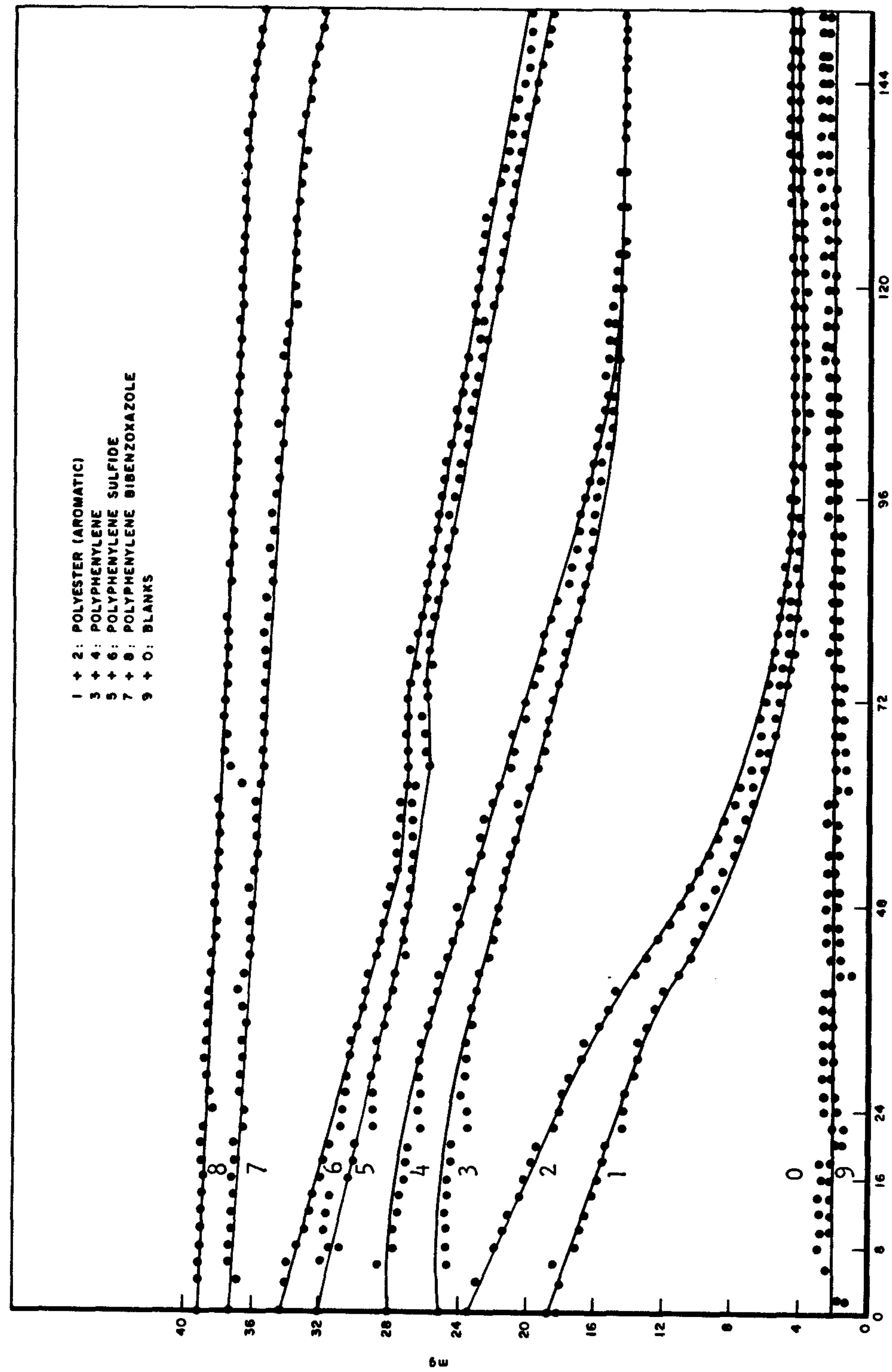

Figure 5. Weight Loss Curves of Several Polymers (II)

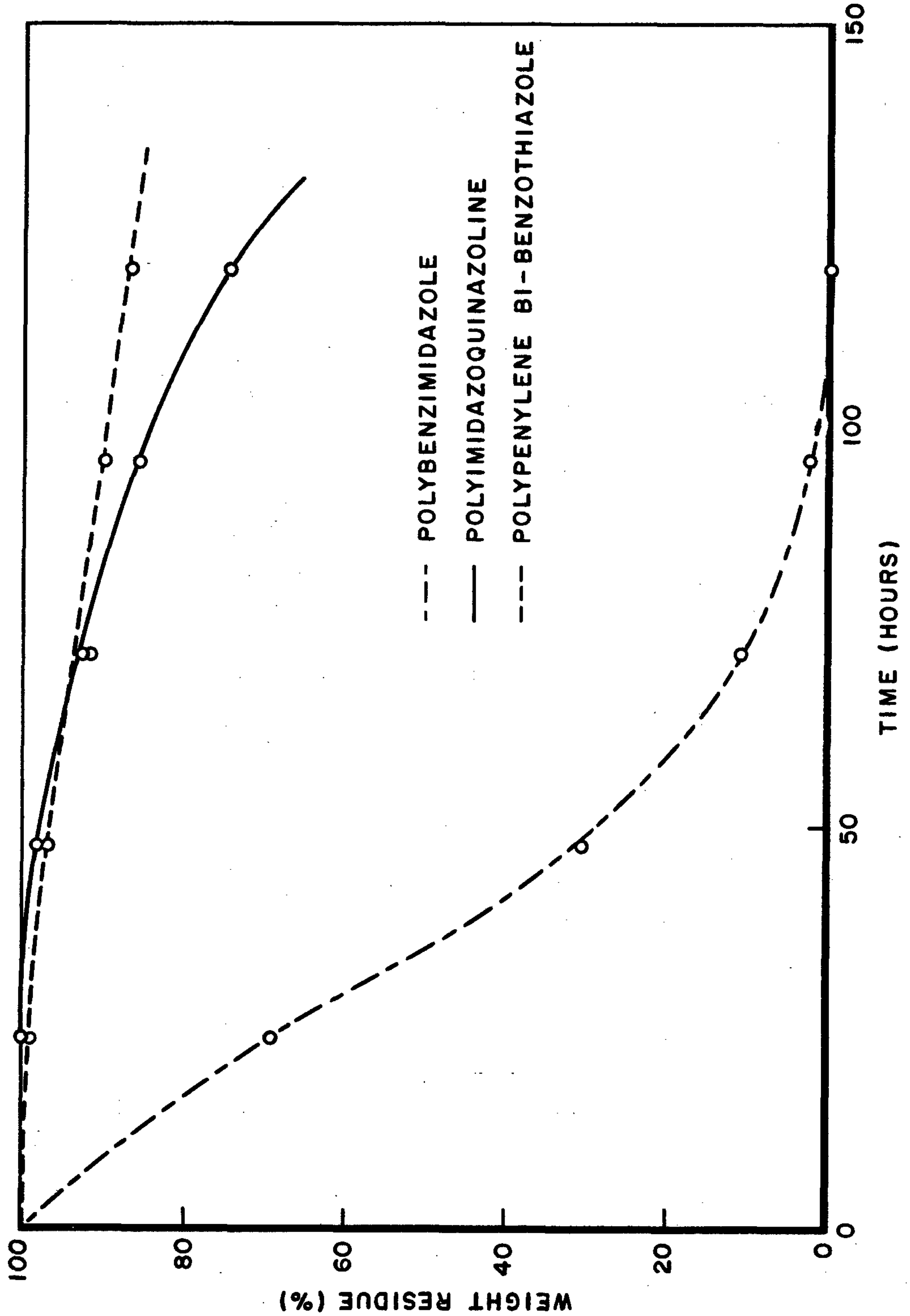

Figure 6. Isothermal Aging (Percent Weight Residue versus Time)

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